IS 7646 (1994): Benzoyl J-acid, Technical [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]

BLANK PAGE

IS 7646 : 1994

(Reaffirmed 2002)

भारतीय मानक

बेंजोइल जे-अम्ल, तकनीकी — विशिष्टि

(पहला पुनरीक्षण)

Indian Standard

BENZOYL J-ACID, TECHNICAL — SPECIFICATION

(First Revision)

UDC 667.21 : 547.554

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

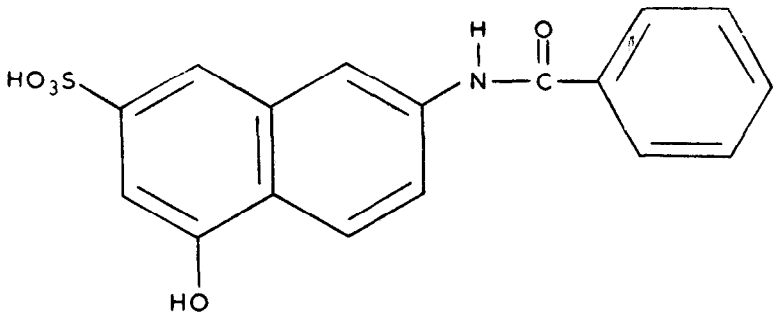
February 1994

Price Group 2

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Benzoyl J-acid ($C_{17}H_{13}O_5NS$) chemically described as 2-benzoylamino-5-naphthol-7-sulphonic acid is an important dye intermediate used in the manufacture of dyes. It has the following structural formula:



BENZOYL J-ACID
(Molecular Mass 343)
C.A.S. Registry Number [132-87-6]

This standard was first published in 1975. The Committee responsible for its preparation decided to update the standard in light of experience gained. In this version, the requirements of Assay and matter insoluble in sodium carbonate have been modified.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

BENZOYL J-ACID, TECHNICAL — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for benzoyl J-acid, technical.

2 NORMATIVE REFERENCES

The following Indian Standards contain provisions which through reference in the text, constitute provisions of this standard. At the time of publication the editions indicated were valid. All standards are subject to revision and parties to agreement, based on the standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
1070 : 1992	Water for general laboratory use (*third revision*)
2552 : 1989	Steel drums (galvanized and ungalvanized) (*third revision*)
5299 : 1969	Methods of sampling and testing for dye intermediates

3 REQUIREMENTS

3.1 Description

The material shall be in the form of a paste, or, if dry, in the form of grey to brown lumps or powder.

3.2 The material shall also comply with the requirements given in Table 1.

Table 1 Requirements for Benzoyl J-Acid, Technical

(*Clauses* 3.2, 5.3.1, 5.3.2 *and* 6.1)

Sl No.	Characteristic	Requirement	Method of Test, Ref to
(1)	(2)	(3)	(4)
i)	Assay, percent by mass (on dry basis), *Min*	70	Annex A
ii)	Matter insoluble in dilute sodium carbonate, percent by mass, *Max*	0·3	10.2 of IS 5299 : 1969
iii)	J-acid content, percent by mass (on dry basis), *Max*	0·5	Annex B

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in steel drums (*see* IS 2552 : 1989) lined with suitable polyethylene film, or as agreed to between the purchaser and the supplier.

4.2 Marking

Each container shall be securely closed and shall bear legibly and indelibly the following information:

a) Name of the material;
b) Indication of the source of manufacture;
c) Net mass of material;
d) Batch or lot number;
e) Month and year of manufacture; and
f) The minimum cautionary notice worded as under:

IT IS A MILD SENSITIZER, LOCAL CONTACT MAY CAUSE DERMATITIS'.

4.2.1 The containers may also be marked with the Standard Mark.

4.2.1.1 The use of the Standard Mark is governed by the provisions of Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5 SAMPLING

5.1 Representative samples of the material shall be drawn as prescribed in 3 of IS 5299 : 1969.

5.2 Number of Tests

5.2.1 Test for assay shall be conducted on each of the individual samples separately.

5.2.2 Tests for the determination for the remaining characteristics, namely, matter insoluble in dilute sodium carbonate solution and J-acid content shall be conducted on the composite sample.

5.3 Criteria for Conformity

5.3.1 *For Individual Samples*

The lot shall be declared as conforming to the requirements of assay if each of the individual test results satisfies the relevant requirement given in Table 1.

5.3.2 *For Composite Samples*

The lot shall be declared as conforming to the requirements of matter insoluble in dilute sodium carbonate solution and J-acid, if the test results satisfy the relevant requirements given in Table 1.

6 TEST METHODS

6.1 Tests shall be conducted according to the methods prescribed and as indicated in col 4 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, 'pure chemicals' and distilled water (*see* IS 1070 : 1992) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[*Table* 1, *Item* (i)]

DETERMINATION OF ASSAY

A-0 OUTLINE OF THE METHOD

The material is dissolved in dilute sodium carbonate solution. A known volume of the solution is coupled with standard 4-chloro-2-anisidine diazo in sodium carbonate medium and from the consumption of diazo, the strength is determined.

A-1 PREPARED SAMPLE

Dry the material at 105 ± 1°C to constant mass. Grind and mix well. Transfer the material to a wide-mouthed bottle and stopper it. Do not expose the sample to an atmosphere containing acidic or alkaline fumes. Use this *prepared sample* for tests.

A-2 REAGENTS

A-2.1 Sodium Carbonate Solution — approximately 10 percent (*m/v*).

A-2.2 Brilliant Yellow Indicator Paper

A-2.3 Phenolphthalein Indicator Paper

A-2.4 Red RC Diazo Solution — 0·1 N.

A-2.5 Alkaline H Acid Indicator Solution

A-2.6 Sodium Chloride — pure.

A-3 PROCEDURE

Weigh accurately about 15 g of the prepared sample (*see* **A-1**) and transfer to a 500-ml beaker with the help of water. Slowly add sodium carbonate solution to get a distinct red test on brilliant yellow paper and a faint test on phenolphthalein paper. Heat if necessary to get a clear solution. Transfer the solution quantitatively to a 500-ml volumetric flask and make up to the mark with water at room temperature. Mix well. Pipette 50 ml aliquot of the above solution into a 1-litre beaker. Add 200-ml ice cold water and 50 ml of sodium carbonate solution. Stir mechanically and cool with washed ice to 0 to 5°C. Titrate with Red RC diazo solution from a cold water-jacketed burette. Near the end point, add 15 g of sodium chloride. Test with alkaline H acid for the excess diazo and with diazo for the coupling component. The end point is reached when the test for the coupling component disappears and a positive test with alkaline H acid is obtained.

A-4 CALCULATION

Assay, precent by mass (on dry basis)

$$= \frac{V}{M} \times 34{\cdot}3$$

where

V = volume in ml of the Red RC diazo solution, and

M = mass in g of the prepared sample taken for the test.

ANNEX B

[*Table* 1, *Item* (iii)]

DETERMINATION OF J-ACID CONTENT

B-0 GENERAL

J-acid is determined by using descending paper chromatographic technique.

B-1 APPARATUS

B-1.1 Chromatographic Sprayer

B-1.2 Developing Chamber

B-1.3 Micropipette

B-2 REAGENTS

B-2.1 Ammonium Hydroxide Solution — 1 percent (*m/v*).

B-2.2 Developer — *n*-propanol mixed with 20 parts by volume of water.

B-2.3 Benzoyl J-Acid — free from J-acid.

B-2.4 J-Acid — pure.

B-2.5 Spray Reagent

0·01 N sulphanilic acid diazo diluted with 40 percent sodium acetate solution (*m/v*) in equal parts at the time of spraying.

B-3 PROCEDURE

First, prepare standard solutions of benzoyl J-acid containing known amounts of J-acid. Into each of three 100 ml volumetric flasks, weigh accurately 1·0 g of benzyl J-acid free from J-acid. Then add 3·0 ml, 4·0 ml and 5·0 ml of 0·10 percent solution of J-acid in 1·0 percent ammonium hydroxide solution to flask No. 1, 2 and 3 respectively. Dissolve the contents of the flask in ammonium hydroxide solution. Thus there shall be 3 solutions of 0·3, 0·4 and 0·5 percent J-acid content. In the fourth flask, weigh about 1·0 g of the prepared sample under test (*see* **A-1**), dissolve in ammonium hydroxide solution and dilute to 100 ml with ammonium hydroxide solution.

B-3·1 Place 10 microlitre spot each of the four solutions using micropipette in the same line to a distance of about 4 cm on filter paper (Whatman No. 1 or equivalent). Place the paper in a descending paper chromatographic glass jar containing the developing reagent and previously saturated with the same developer. Allow the solvent to run in a descending manner for about 30 cm from the spot. This will take about 12 hours. Take out the paper after 30 cm run and dry the solvent completely. Then spray the paper with spray reagent. After 10 minutes of spray, compare the intensity of the J-acid spot visually with those of known standards.

B-4 CALCULATION

Report J-acid content as that which is nearest in intensity to the standard. In case the colour intensity does not come in the range of standard spots, repeat the whole procedure using different percentages of J-acid.

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act, 1986* to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such a review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Handbook' and 'Standards Monthly Addition'.

This Indian Standard has been developed from Doc: No. PCD 11 (1150)

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones : 331 01 31, 331 13 75

Telegrams : Manaksanstha
(Common to all Offices)

Regional Offices:	Telephones
Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002	331 01 31, 331 13 75
Eastern : 1/14 C.I.T. Scheme VII M, V.I.P. Road, Maniktola, CALCUTTA 700054	37 84 99, 37 85 61, 37 86 26, 37 86 62
Northern : SCO 445-446, Sector 35-C, CHANDIGARH 160036	53 38 43, 53 16 40, 53 23 84
Southern : C.I.T. Campus, IV Cross Road, MADRAS 600113	235 02 16, 235 04 42, 235 15 19, 235 23 15
Western : Manakalaya, E9 MIDC, Marol, Andheri (East) BOMBAY 430093	632 92 95, 632 78 58, 632 78 91, 632 78 92

Branches : AHMADABAD. BANGALORE. BHOPAL. BHUBANESHWAR. COIMBATORE. FARIDABAD. GHAZIABAD. GUWAHATI. HYDERABAD. JAIPUR. KANPUR. LUCKNOW. PATNA. THIRUVANANTHAPURAM.

Printed at Paragon Enterprises, Delhi, India